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12. ABSTRACT (Maximum 200 words) Metal containing cluster molecules are produced in the gas phase and studied with mass spectrometry and laser spectroscopy. These systems are evaluated to explore the fundamental interactions between metal atoms and small molecules. These studies probe metal van der Waals bonding, metal-metal bonding and related interactions likely to be encountered when metals are condensed in cryogenic matrices in the form of metal seeded fuels. Additional studies probe new gas phase metal carbide and metal oxide clusters important as potential ceramic materials precursors.				
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**- Final Report -**

**For the Period December 1, 1993 to November 30, 1996**

**CLUSTER MODELS OF METAL SEEDED ENERGETIC MATERIALS**

**AFOSR Contract No. F49620-94-1-0063**

**January 1997**

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## OBJECTIVES

The goal of this project is to prepare and characterize novel molecules containing metals which are relevant for the understanding of prospective high energy propellant systems. Two classes of molecules are studied: 1) weakly bound van der Waals complexes containing light metal atoms (Li, Al, Mg) and 2) mixed metal diatomics of these same light elements (LiB, LiAl, AlB, AlMg, etc.). Both of these classes of molecules are prepared and studied in supersonic molecular beams, and they are characterized with laser spectroscopy and time-of-flight mass spectroscopy. In a related area, we are also investigating metal compound clusters (transition metal carbides and oxides) which exhibit novel patterns of cluster growth, and which may have applications as microparticle ceramic materials.

## STATUS OF EFFORT

A variety of weakly bound complexes have been prepared and characterized containing aluminum atoms bound by van der Waals forces to rare gas atoms or small molecules. Threshold photoionization has been applied to  $\text{Al}-(\text{CO}_2)_x$  and  $\text{Al}-(\text{N}_2)_x$ . Attempts to extend these studies to  $\text{Al}-(\text{H}_2)_x$  complexes or  $\text{Li-R}_2$  complexes have been thusfar unsuccessful. High resolution photoelectron spectroscopy has been applied to the Al-Ar and Al-Kr complexes, and these are the only metal complexes to be studied with this technique in any laboratory, but attempts to extend these studies to other complexes have also been unsuccessful. Our efforts to make new metal dimers and new metal compound clusters have been far more productive. Both of these experiments are made possible by the development of new techniques for mixed metal and metal

composite sample preparation. Using this new methodology, we have been able to generate mixed clusters of lithium and magnesium, and we have measured the first spectroscopy for the diatomic LiMg. Samples have now been prepared to enable spectroscopic searches for AlMg, MgC, AlB, etc. The same kind of composite sample preparation has allowed the production and characterization of several new metal carbide cluster systems. We have measured the first ionization potentials for  $M_8C_{12}$  "met-car" clusters and compared these results to recent theoretical calculations. We have also produced bismuth and antimony oxides (both negative and positive ions), which exhibit striking magic number patterns.

## ACCOMPLISHMENTS/NEW FINDINGS

### Metal Dimers

The production and spectroscopy of metal dimers such as LiB, LiMg and AlB is difficult because these elements do not exist as compounds or alloys in readily available solid form. We have therefore designed and constructed a vapor deposition apparatus, using a metal oven source, to produce films of one metal component on the surface of a solid rod sample of the other component. This deposition apparatus has made it possible to produce rod samples of lithium on aluminum, lithium on magnesium, lithium on BN ceramic, etc. Laser vaporization of these samples penetrates through the thin surface film to the underlying metal, producing both components in the gas phase where mixed molecules can form.

The initial success of this methodology has resulted in the first spectroscopy of LiMg. To make this molecule, we vaporized a composite sample which had a thin film of lithium deposited

on a solid rod of magnesium. We have measured spectra for two electronic band systems, obtaining vibrational frequencies and extrapolated dissociation energies for the excited states. Isotope shifts in the vibronic levels makes it possible to assign the electronic origins for these states. These states are easily correlated to the  $\text{Li}(3p)(^2P) + \text{Mg}(^1S)$  atomic asymptote, making it possible to determine the ground state dissociation energy for the molecule, which is  $3400\text{ cm}^{-1}$ . This value is in good agreement with theory. The low dissociation energy is understandable because the bonding results from the  $\text{Li } 2s^1 + \text{Mg } 3s^2$  configuration, which produces a  $\sigma^2\sigma^{*1} (^2\Sigma)$  ground state and a bond order of  $1/2$ . The spectroscopic constants determined for LiMg are shown in the table below. LiMg ( $D_0=3434\text{ cm}^{-1}$ ;  $0.43\text{ eV}$ ) is less strongly bound in its ground electronic state than  $\text{Li}_2$  ( $D_0=1.04\text{ eV}$ ), but much more strongly bound than  $\text{Mg}_2$  ( $D_0=0.05\text{ eV}$ ). It is difficult to form, but a reproducible spectrum can be obtained.

Table 1. Spectroscopic constants for LiMg. All units are  $\text{cm}^{-1}$ .

state	$\nu_{00}$	$\omega_e$	$D_0$
$X^2\Sigma$	-	191.8	3434
C	30,370	241.2	3989
D	31,161	255.9	3199

## Metal Oxide Clusters

We are searching for interesting new metal compound systems which may produce stable clusters useful as precursors for ceramic materials preparation. This investigation includes metal oxides and metal carbides, etc. We have recently obtained interesting new "magic number" mass spectra for metal oxide clusters of antimony and bismuth in both the positive and negative ion modes of our mass spectrometer. The species  $M_3O_4^+$ ,  $M_4O_5^+$ ,  $M_5O_7^+$ ,  $M_6O_8^+$ ,  $M_7O_{10}^+$ ,  $M_9O_{14}^+$  and  $M_{11}O_{17}^+$  are produced with especially enhanced abundance for both antimony and bismuth. Figure 1 shows the mass distributions of Bi/O cluster cations produced. At each cluster size, there is a unique oxide stoichiometry produced. In negative ions, the oxide clusters have only been observed at smaller sizes so far, but there is also a unique oxide stoichiometry which is the same for antimony and bismuth, but which is different from the corresponding cation with the same number of metal atoms (e.g.  $M_2O_5^-$ ). These observations imply that there is a strong electronic structure effect working to produce stable clusters, rather than a geometric effect. Otherwise, there would not be such a dramatic change in the cluster stoichiometry for different charge states. For many of these systems, we are able to build model cage structures which have fully satisfied valence for metal and oxygen. Some of these proposed structures are shown in Figure 2. We have contacted theoretical research groups to secure some additional justification for these proposed structures.

We have also studied these same metal oxide clusters with multiphoton ionization at 193 nm (ArF excimer laser), and the abundant cations produced by this process are the same as those produced when cations grow directly. We have mass selected these cations and photodissociated them at 355 nm, and the fragments produced are smaller cations which were themselves magic number species in the growth distribution. As an example, Figure 3 shows the photodissociation of

$\text{Bi}_7\text{O}_{10}^+$  at 355 nm. The fragment ions produced are  $\text{Bi}_5\text{O}_7^+$  and  $\text{Bi}_3\text{O}_4^+$ , which are also evident in Figure 1 as magic number cations.

### Metal Carbide Clusters

We have also made progress in the study of metal carbide clusters and in the production of new metal carbide clusters. In previous work we had documented interesting mass spectral patterns and photodissociation behavior of metal carbide clusters of the early transition metals (Ti, V, Zr, etc.). These species were produced by vaporizing solid metal with gaseous hydrocarbons added to the plasma. As shown first by Castleman, the reactivity of the early transition metals is great enough so that insertion chemistry takes place with elimination of hydrogen, producing pure metal-carbides. While carbides of the late transition metals may also be interesting, and they may be intrinsically stable, they cannot be formed by this plasma chemistry because the metals are less reactive. Plasma chemistry reactions for these metals lead to addition to hydrocarbon species in the gas phase without elimination of hydrogen, or to no metal carbide clusters at all. We have therefore employed our vapor deposition method, using metal films deposited on graphite rods, to produce metal-carbide samples without the presence of hydrogen. Vaporization of these samples produces the desired metal carbide clusters in many cases. For example, carbides of antimony or bismuth cannot be produced by the hydrocarbon plasma chemistry method, but they are produced readily from composite sample (metal film on carbon rod) vaporization. Another technique we have used with success is *electrochemical* film deposition. Standard electrochemical preparations make it possible to deposit thin films of many transition metals on carbon rods. We have used this approach to produce composite samples of carbon coated with silver, copper, cobalt and nickel. In

each case, vaporization experiments produce metal carbide clusters in the gas phase. We have found that each system requires a different metal film thickness for best cluster production. Depending on this variable, the metal carbide clusters produced may be rich in carbon, like the nickel system shown in the figure, or they may be rich in metal. We have not found any metal which does not produce carbide clusters. However, we have not yet found the dramatic magic number patterns for later transition or main group metal carbides like those observed for the early transition metals. Additional experiments to optimize cluster production by variation of the film thicknesses, and to explore photodissociation of these new systems are underway. We have used similar methods to produce metal silicide clusters from films deposited on a silicon rod.

In a final area of progress, we have continued the characterization of magic number metal carbides of the early transition metals, where the 8/12 M/C stoichiometry is produced with extraordinary abundance. We have used tunable ultraviolet lasers to obtain threshold photoionization of these species in the limit of extremely low laser power (to eliminate multiphoton effects). We find that  $\text{Ti}_8\text{C}_{12}$  has an ionization potential of 4.9 eV, which seems to be an extremely low value for a closed shell system.  $\text{V}_8\text{C}_{12}$ ,  $\text{Zr}_8\text{C}_{12}$  and  $\text{Nb}_8\text{C}_{12}$  all have ionization potentials greater than our highest tunable laser energy (5.7 eV). The low IP of  $\text{Ti}_8\text{C}_{12}$  is consistent with the predictions of some theoretical studies, but the trend for the other metal carbides (all having a greater IP's than  $\text{Ti}_8\text{C}_{12}$ ) is not consistent with the predictions of any theoretical study. This is the first direct measurement of a physical property of met-cars clusters which has been calculated by theory, and the discrepancy illustrates the difficulties of theory on such systems.



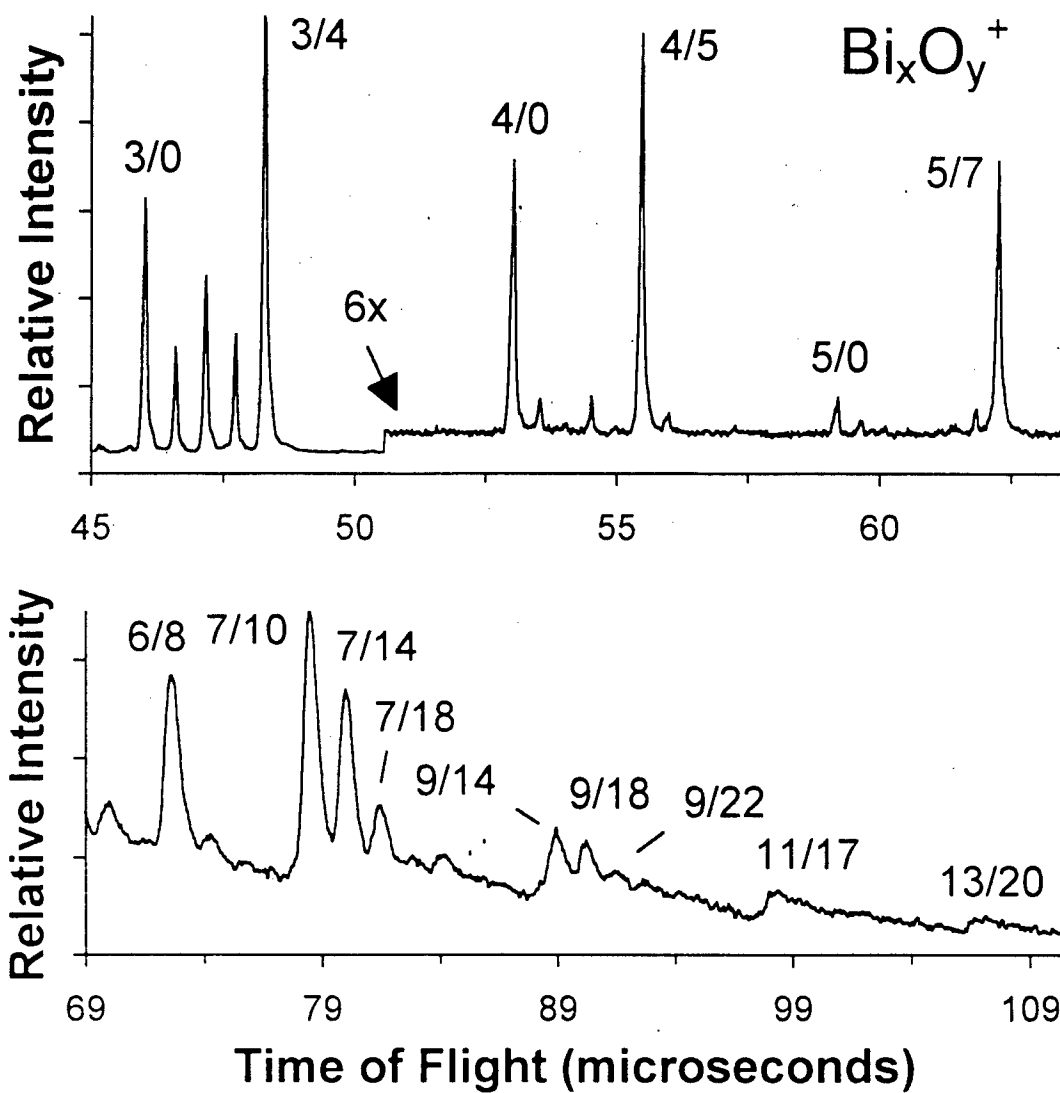
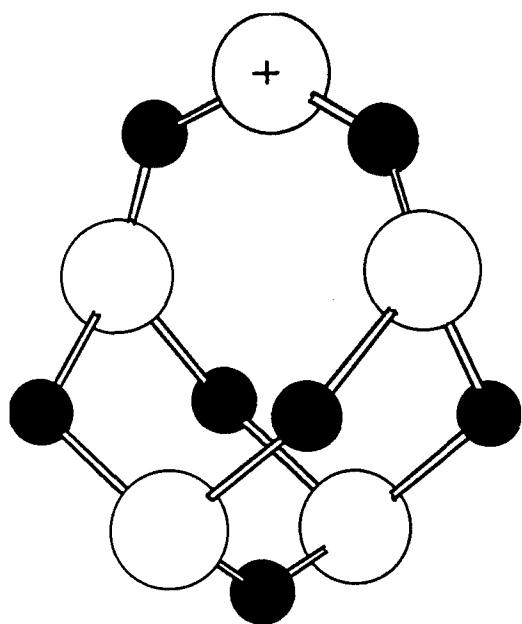
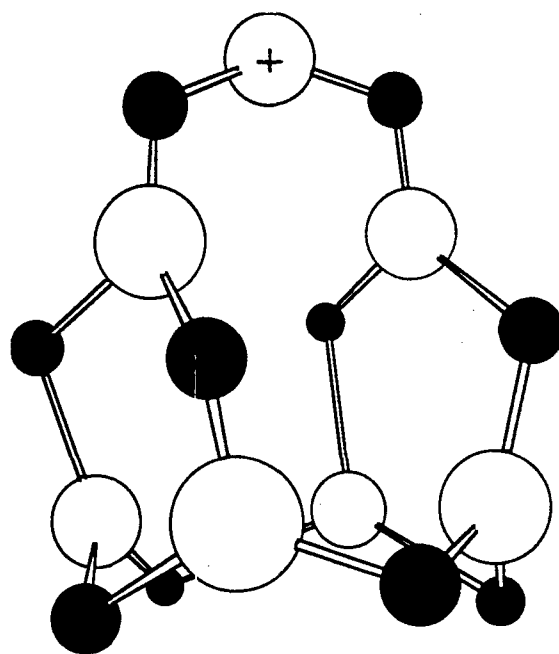


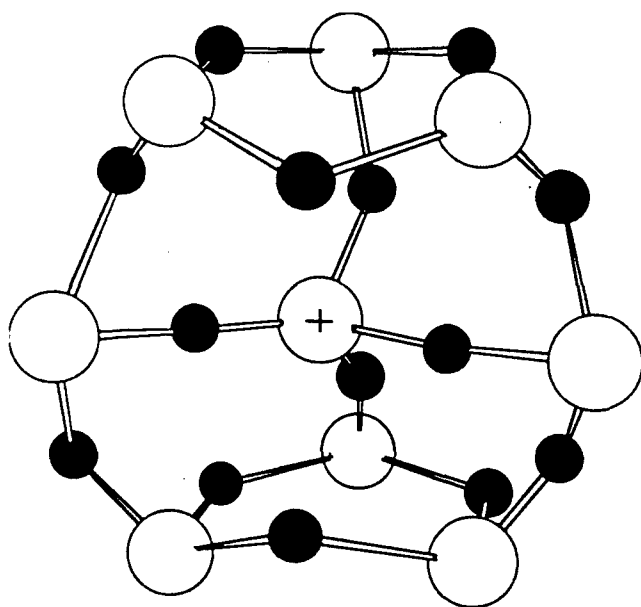
Figure 1.



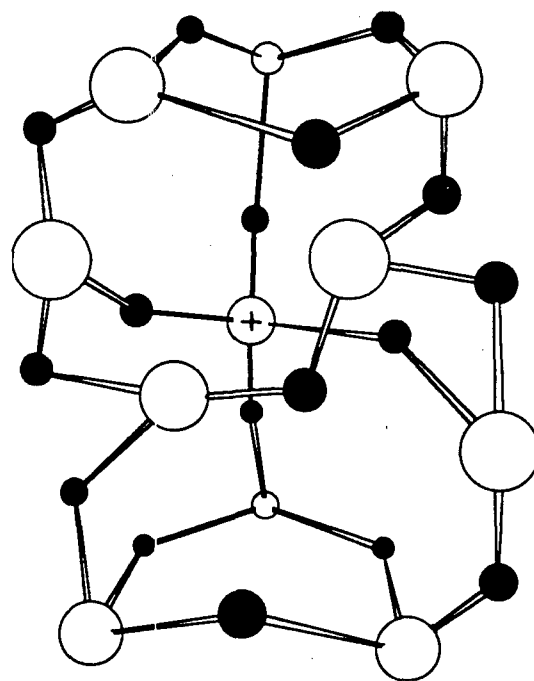
a)  $\text{Bi}_5\text{O}_7^+$



b)  $\text{Bi}_7\text{O}_{10}^+$



c)  $\text{Bi}_9\text{O}_{14}^+$



d)  $\text{Bi}_{11}\text{O}_{17}^+$

$\text{Bi}_7\text{O}_{10}^+$   
355 nm

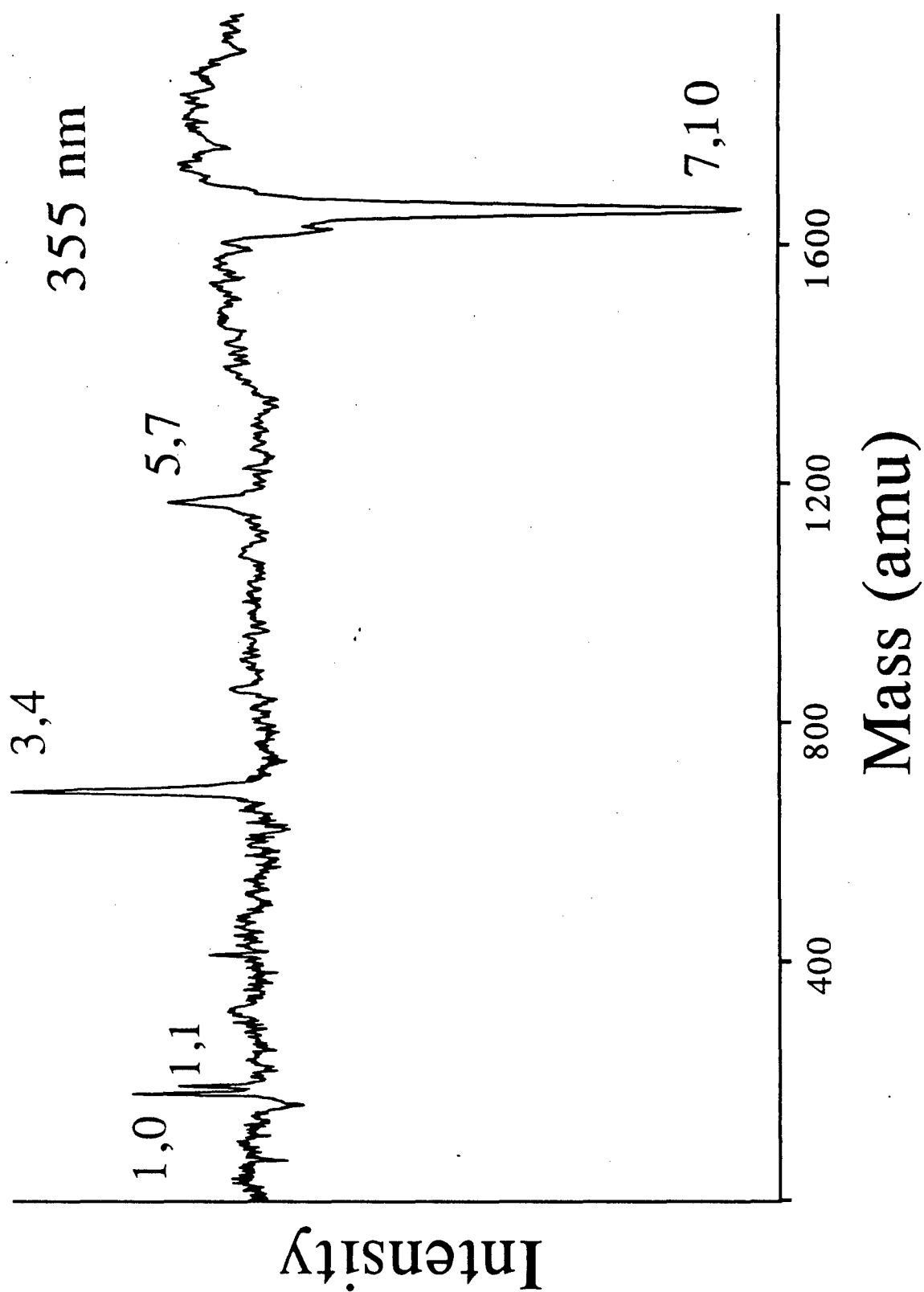


Figure 3.

## PERSONNEL SUPPORTED BY AND/OR ASSOCIATED WITH PROJECT

### Faculty:

Professor Michael A. Duncan (one month summer salary)

### Graduate Students:

Student	Degree Program	Social Security Number
Lori R. Brock	Ph.D. (rec'd 6/96)	[REDACTED]
Kenneth R. Berry	Ph.D.	[REDACTED]
Steve Pullins	Ph.D.	[REDACTED]
Michele France	M.S.	[REDACTED]
John Reddic	Ph.D.	[REDACTED]
Areatha Knight	M.S.	[REDACTED]

### Undergraduate Student:

Jeff Tucker	B.S.	[REDACTED]
Jason Robinson	B.S.	[REDACTED]

### Postdoctoral Fellows:

Dr. Chris Scurlock	[REDACTED]
Dr. Andreas Stangassinger	[REDACTED]

## PUBLICATIONS IN THIS FUNDING PERIOD

1. K.F. Willey, D.L. Robbins, C.S. Yeh and M.A. Duncan, "Cluster Ion Photodissociation and Spectroscopy in a Reflectron Time-of-Flight Mass Spectrometer," *ACS Symposium Series 549: Time-of-Flight Mass Spectrometry*, R.J. Cotter, ed., American Chemical Society, Washington, DC, 1994, 61 (invited).
2. C.S. Yeh, K.F. Willey, D.L. Robbins and M.A. Duncan, "Photodissociation of Magnesium Ion-Molecule Complexes in a Reflectron Time-of-Flight Mass Spectrometer," *Intl. J. Mass Spectrom. and Ion Processes* **131**, 307 (1994) (invited).
3. J.S. Pilgrim, K.R. Berry, and M.A. Duncan, "Photodissociation Spectroscopy of Magnesium Ion-Rare Gas Complexes," *J. Chem. Phys.* **100**, 7945 (1994).
4. C.S. Yeh, J.S. Pilgrim, D.L. Robbins, K.F. Willey and M.A. Duncan, "Spectroscopy of Weakly-Bound Magnesium Ion Complexes," *Intl. Rev. Phys. Chem.* **13**, 231 (1994) (invited).
5. L.R. Brock, J.S. Pilgrim and M.A. Duncan, "Photoionization Electronic Spectroscopy of LiAl," *Chem. Phys. Lett.* **230**, 93 (1994).
6. D.L. Robbins, L.R. Brock, J.S. Pilgrim and M.A. Duncan, "Electronic Spectroscopy of the  $Mg^+-N_2$  Complex: Evidence for Photoinduced Activation of  $N_2$ ," *J. Chem. Phys.* **102**, 1481 (1995).
7. L.R. Brock and M.A. Duncan, "Threshold Photoionization of  $Al-(N_2)_x$  and  $Al-(CO_2)_x$  Complexes: Evidence for Solvation-Induced Reactions," *J. Phys. Chem.* **99**, 16,571 (1995).
8. L.R. Brock and M.A. Duncan, "Near-Threshold Photoionization to Probe Neutral "Met-Cars" Clusters," *J. Phys. Chem.* **100**, 5654 (1996).
9. L.R. Brock, J.S. Pilgrim, D.L. Robbins and M.A. Duncan, "A Convenient Modification to the Newport Pulsed Molecular Beam Valve," *Rev. Sci. Instr.* **67**, 2989 (1996).
10. J.E. Reddic and M.A. Duncan, "Composite Samples and the Generation of New Metal Carbide Clusters," *Chem. Phys. Lett.*, in press.
11. M.A. Duncan, "Spectroscopy of Metal Ion Complexes: Gas Phase Models for Solvation," *Ann. Rev. Phys. Chem.* **48**, xxxx (1997), in press (invited).
12. M.R. France, J.W. Buchanan, J.C. Robinson, S.H. Pullins, J.L. Tucker and M.A. Duncan, "Antimony and Bismuth Oxide Clusters: A New Family of Magic Number Clusters," *J. Am. Chem. Soc.*, in preparation.

13. K.R. Berry and M.A. Duncan, "Photoionization Spectroscopy of LiMg," *Chem. Phys. Lett.*, in preparation.

## INTERACTIONS/TRANSITIONS

### a) PRESENTATIONS

#### Invited Lectures Presented on this Research

1. "Spectroscopy of Gas Phase Clusters," *Chemistry Colloquium*, University of Alabama-Birmingham, January 1994.
2. Spectroscopy and Photodissociation Dynamics of Metal Cluster Ions," *Physical Chemistry Seminar*, University of California-Irvine, January 1994.
3. "Photodissociation and Photoionization Spectroscopy of Metal Clusters," *SPIE Meeting of the Optical Society of America*, Los Angeles, January 1994.
4. "Spectroscopy and Photochemistry of Metal Cluster Ions," *Physical Chemistry Seminar*, Argonne National Labs, Argonne, IL, February 1994.
5. "Spectroscopy and Photochemistry in Metal Cluster Ions," *Physical Chemistry Seminar*, Northwestern University, February 1994.
6. "Photodissociation of Metal Cluster Ions: From Solvated Ions to Nanocrystals," *Departmental Colloquium*, University of Iowa, February 1994.
7. "Spectroscopy and Photochemistry in Metal Cluster Ions," *Physical Chemistry Seminar*, Iowa State University, February 1994.
8. "Photodissociation Dynamics in Metal Ion-Molecule Cluster Complexes," *Joint Japanese American Cluster Workshop*, Oahu, Hawaii, March 1994.
9. "Photoinduced Chemistry in Metal Ion Cluster Complexes," *Symposium on Cluster Models of Condensed Phased Chemistry*, National Meeting of the American Chemical Society, San Diego, CA, March 1994.
10. "Metal-Carbon Cages and Nanocrystals," *Symposium on Novel Materials*, National Meeting of the American Chemical Society, San Diego, CA, March 1994.
11. "Metal-Carbon Cages and Nanocrystals," *Symposium on Novel Forms of Carbon*, Spring Meeting of the Materials Research Society, San Francisco, April 1994.

12. "Spectroscopy and Photodissociation of Metal Cluster Ions," *Physical Chemistry Seminar*, University of California-Davis, April 1994.
13. "Photodissociation and Spectroscopy of Metal Cluster Ions," *Physical Chemistry Seminar*, University of Delaware, May 1994.
14. "Metal-Carbon Cages and Nanocrystals," *Fullerene Symposium, National Meeting of the Electrochemical Society*, San Francisco, May 1994.
15. "Resonance Enhanced Photodissociation Spectroscopy of Metal Ion Cluster Complexes," *Gordon Conference on Multiphoton Processes*, New London, NH, June 1994.
16. "Photodissociation and Photoionization Spectroscopy of Metal Clusters," *Physical Chemistry Seminar*, University of North Carolina-Chapel Hill, September 1994.
17. "Photoionization and Photodissociation Spectroscopy of Metal Clusters and Metal Complexes," *Physical Chemistry Seminar*, Arizona State University, October 1994.
18. "Spectroscopy of Metal Ion Complexes," International Laser Science Conference (ILS-X), *Symposium on Novel Techniques for Cluster Spectroscopy*, Dallas, TX, October 1994.
19. "Cluster Models of Energetic Materials," *Symposium on High Energy Materials*, Southeast Regional Meeting of the American Chemical Society, Birmingham, AL, October 1994.
20. "Photodissociation Spectroscopy of Small Metal Clusters," *Chemical Physics Seminar*, California Institute of Technology, November 1994.
21. "The Chemistry of Microclusters," *EΦΔ Science Club Colloquium*, Oakwood College, Huntsville, AL, January 1995.
22. "Photoionization and Photodissociation of Metal Clusters," *McGill Chemical Society Lecture*, McGill University, Montreal, Canada, January 1995.
23. "Photodissociation and Threshold Photoionization Studies to Probe Metal Cluster Ions," *Gordon Conference on the Structure, Energetics and Reaction Dynamics of Gaseous Ions*, Ventura, CA, February 1995.
24. "Photodissociation of Metal Clusters: From Solvated Ions to Nanocrystals," *Chemistry Department Colloquium*, University of Puerto Rico, March 1995.
25. "Photodissociation Studies of Metal Clusters and Metal Ion Complexes," *Analytical/Physical Chemistry Seminar*, Texas A&M University, April 1995.

26. "Photodissociation Studies of Metal Clusters and Metal Ion Complexes," *Analytical/Physical Chemistry Seminar*, University of Texas-Austin, April 1995.
27. "Photodissociation and Photoionization Spectroscopy of Metal Complexes," *Yamada Conference on Cluster Structures and Dynamics*, Shimoda, Shizuoka, Japan, May 1995.
28. "Photoionization and Photodissociation Spectroscopy of Weakly Bound Metal Complexes," *Physical Chemistry Seminar*, University of Bern, Switzerland, September 1995.
29. "Photoionization and Photodissociation Spectroscopy of Weakly Bound Metal Complexes," *Chemical Physics/Condensed Matter Physics Seminar*, University of Chicago, October 1995.
30. "Synthesis and Spectroscopy of Gas Phase Metal Clusters," *Departmental Colloquium*, Western Carolina University, November 1995.
31. "Gas Phase Clusters: From Nanocrystals to Solvated Metal Ions," *Physical Chemistry Seminar*, Yale University, December 1995.
32. "Gas Phase Metal Clusters: From Nanocrystals to Solvated Metal Ions," *Departmental Colloquium*, University of South Carolina, December 1995.
33. "Photodissociation Spectroscopy of Metal Ion Complexes," *American Society for Mass Spectrometry Winter Meeting*, Sanibel Island, FL, January 1996.
34. "Solvation of Metal Atoms in Gas Phase Clusters: Spectroscopy and Chemistry," *Chemical Physics Seminar*, Pacific Northwest National Laboratory, Richland, WA, February 1996.
35. "Tandem Mass Spectrometry with Lasers and Time-of-Flight Spectrometers," *Athens-Atlanta Mass Spectrometry Group*, Athens, GA, March 1996.
36. "Spectroscopy of Metal Ion Cluster Complexes," *Gordon Conference on Molecular and Ionic Clusters*, Barga, Italy, May 1996.
37. "Photoionization and Photodissociation Studies of Metal Carbide Clusters," *70th Colloid and Surface Science Symposium*, Clarkson University, Potsdam, NY, June 1996.
38. "Spectroscopy of Weakly Bound Metal Complexes," *Symposium on Bimolecular Interactions of Free Radicals, National Meeting of the American Chemical Society*, Orlando, FL, August 1996.
39. "Photodissociation of Gas Phase Metal Clusters: From Nanocrystals to Solvated Cations," *Departmental Colloquium*, Baylor University, October 1996.



40. "Photodissociation of Gas Phase Metal Clusters: From Nanocrystals to Solvated Cations," *Departmental Colloquium*, Virginia Commonwealth University, October 1996.
41. "Electrostatic Bonding in Metal Atom Complexes," *Waterloo Chemical Physics Symposium*, Waterloo, Canada, November 1996.

#### Poster and Contributed Presentations of this Research

1. D.L. Robbins, J.S. Pilgrim and M.A. Duncan, "Photodissociation Spectroscopy of  $\text{Mg}^+-\text{N}_2$ ," *Ohio State International Symposium on Molecular Spectroscopy*, Columbus, Ohio, June 1994.
2. "Photodissociation Studies of Metal-Carbon Clusters and Nanocrystals," *AFOSR Conference on Chemical Dynamics and New Materials*, Washington, DC, October 1994.
3. M.A. Duncan, "Metal Dimers and Metal Complexes: Models for Metal-Seeded Energetic Fuels," *AFOSR High Energy Density Materials Contractor's Meeting*, Wood's Hole, MA, June 1995.
4. C.T. Scurlock and M.A. Duncan, Rotationally Resolved Photodissociation Spectroscopy of  $\text{Mg}^+-\text{Ar}$ ," *Ohio State International Symposium on Molecular Spectroscopy*, Columbus, Ohio, June 1995.
5. L.R. Brock and M.A. Duncan, "Near-Threshold Photoionization of Metal-Carbide Clusters," *Gordon Conference on Metal and Semiconductor Clusters*, New London, NH, August 1995.
6. L.R. Brock, A.M. Knight and M.A. Duncan, "Mass Analyzed Threshold Ionization of Aluminum Atom Complexes," *European Science Foundation Meeting on High Resolution Spectroscopy with Photoelectrons*, Lenggries, Germany, September 1995.
7. M.A. Duncan, "Photodissociation and Photoionization of Metal Compound Clusters," *AFOSR Molecular Dynamics Program Contractor's Meeting*, Boulder, CO, June 1996.

## **b) CONSULTATIVE AND ADVISORY FUNCTIONS**

None

## **c) TRANSITIONS**

Our measurements of the spectroscopy and dissociation energies of magnesium cation ion-molecule complexes have been used by researchers at the Air Force Philips Laboratory, Hanscom AFB, in their analysis of atmospheric chemistry of  $Mg^+$ . In particular, our demonstration that  $Mg^+-N_2$  is a relatively stable molecule provides a possible explanation for anomalous low concentrations of  $Mg^+$  in the ionosphere (W.J. McNeil, S.T. Lai and E. Murad, *J. Geophys. Res.* **101**, 5251 (1996)).

## **NEW DISCOVERIES, INVENTIONS OR PATENT DISCLOSURES**

None

## **HONORS/AWARDS**

Appointment as University Research Professor, University of Georgia, May 1995.